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DATE: Monday, September 30, 2002

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DB=USPT,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=OR

L3	L1 and (calcium adj1 \$phosphate)	4	L3
L2	L1 and (liposome\$)	0	L2
L1	(implant or implants) same (electrolysis or electrolit\$)	40	L1

END OF SEARCH HISTORY

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L3: Entry 4 of 4

File: USPT

May 10, 1994

DOCUMENT-IDENTIFIER: US 5310464 A

TITLE: Electrocrystallization of strongly adherent brushite coatings on prosthetic alloys

Abstract Text (1):

An electrolytic method for providing bone-emulating, phosphate coatings on prosthetic appliances. Such coatings serve to enhance bone fixation after implantation of the appliances. The method of the invention is an electrolysis process wherein the appliance to be coated is immersed in a phosphate-containing electrolyte to serve as the cathode of the electrolysis process. When current is applied to the electrolysis cell, the electrolyte solution, which includes calcium ions and dihydrogen phosphate ions, is caused to rapidly increase in (localized) pH proximate the cathode element. The localized pH increase creates a supersaturated local condition causing less soluble calcium phosphate salts to crystallize out of the electrolyte solution and adhere to the cathode, thus effecting thereon a coating of brushite.

Brief Summary Text (5):

Although the mechanism by which fixation of bone to a transplant occurs is somewhat beyond the scope of the instant disclosure, it has been observed that the coating of metallic prostheses with phosphate ceramics has received a great deal of recent attention because of the apparent propensity of these coatings to accelerate bone fixation during the early stages following implantation. Current articles in the literature have given ample reason to believe that the rate of metal ion release from some alloys can be reduced by calcium phosphate coatings. Further, reviews concerning applications of hydroxyapatite coatings on metallic implants were given significant treatment by Ducheyne, P.; Lemons, J.E.; Eds.; "Bioceramics: Material Characteristics Versus In Vivo Behavior", New York, The New York, Academy of Sciences, 1988.

Brief Summary Text (6):

The most frequently used means or process for the deposition of calcium phosphate materials on prosthetic alloys is by way of plasma or flame spraying. There was recently reported by Takayuki Shimamune and Masashi Hosonuma in Chemical Abstracts, volume 109: 11784d and volume 109:11785e, data on a calcium phosphate-coated medical composite implant material and a process for its manufacture. Therein, it is indicated that a calcium phosphate-coated compatible composite material, comprising a metallic substrate and an oxide layer of more than one metal selected from the group consisting of titanium, zirconium, hafnium, vanadium, niobium, tantalum, tin, cobalt, aluminum, chromium, molybdenum and tungsten is overlaid with a layer of calcium phosphate which can be produced by plasma or flame spraying. This composite has an affinity for the tissue of bone or teeth and finds its use as an implant material for artificial bone, teeth and teeth roots, or as a bonding material for such implant materials.

Brief Summary Text (7):

Also significant, and as disclosed in the Chemical Abstracts references are European Patent applications 0,264,353 and 0,264,354, both made by Shimamune and Hosonuma. These disclosures teach a composite material and a process for the production thereof which comprises a metallic substrate having thereon an oxide layer, the oxide layer consisting essentially of the oxide of one or more metals denoted in the previously mentioned group, and thereafter a calcium phosphate overlay on the oxide layer. The composite is made by oxidizing a metallic substrate, either thermally or

electrolytically, to form a layer of the oxide or the metallic substrate component alone or a layer of mixed oxide of the metallic substrate component and a metal component in the electrolyte. Alternatively, heating of the metallic substrate is accomplished to stabilize the surface thereof; and, then a coating of calcium phosphate compound is formed on the surface. Essentially, the Shimamune et al methodology comprises two distinct processes. The first, the perhaps preferred embodiment, is a process used to produce a calcium phosphate compound-coated composite material suitable as an implant material. This comprises thermally oxidizing a metallic substrate to form, on the surface of the metallic substrate, a layer of the oxide of the metallic substrate component, such providing excellent corrosion resistance in the living body. Then, there is formed on the oxide coating a layer of calcium phosphate compound such as apatite hydroxide, which has been determined to have affinity to the living body on the surface of the aforementioned oxide layer. The second Shimamune et al. embodiment is a process for producing a calcium phosphate compound-coated composite material, also suitable as an implant, which comprises electrolyzing a metallic substrate in an electrolyte to form on the surface thereof a coating of the oxide of the metallic substrate component alone or a mixed oxide of the metallic substrate component and a metal component of the electrolyte. Thereafter, as in the first embodiment, there is formed on the oxide, a calcium phosphate coating such as apatite hydroxide.

Brief Summary Text (8):

When the metallic substrate is made of stainless steel or a cobalt-chromium alloy, unlike the case wherein the metallic substrate is made of titanium or a titanium alloy, it is necessary to become highly selective in the electrolyte usage. Shimamune et al. teach that, if anode polarization is carried out in an acidic solution, the metal surface is dissolved and the desired oxide layer becomes difficult to obtain. Continuing, they teach that, in a strongly alkaline solution, the oxide on the surface of the metallic substrate is partially dissolved and thus, in some cases, a sufficiently grown oxide layer cannot be obtained. The limitation which is therefore placed on the process is that it becomes necessary to choose an electrolyte having a pH of 6 to 13. Quite matter-of-factly, Shimamune et al. insist that the method of forming the calcium phosphate coating and the conditions under which the method is carried out are not critical.

Drawing Description Text (3):

FIGS. 1A and 1B are scanning electron micrographs of electrolytically deposited calcium phosphate coatings on 316L stainless steel;

Detailed Description Text (2):

Given the number of arthroplasties performed yearly, the need for a physically stable, biocompatible material which can be easily deposited on implant surfaces is considerable. It has been recently reported, in a prominent medical journal, that an estimated 120,000 total hip implants are required yearly in the United States. As early as 1986, it was estimated that, by 1990, the annual joint prostheses requirements would reach 500,000. Thus, a process for economically coating a metallic prostheses with phosphate ceramic, notably the various calcium phosphate coatings, becomes compelling when viewed in the light of the ability of these coatings to accelerate bone fixation during the early recuperative stages after implantation.

Detailed Description Text (7):

A cathode is prepared by using a device (prosthesis) comprised of 316L stainless steel. To form the deposit depicted in FIG. 1A, the electrolysis is run over a period of 2 hours 13 minutes at 1 mA/cm.² for a total of 8 C/cm.². In FIG. 1B, a calcium phosphate deposit at 10 mA/cm.² resulted from a total of 5 C/cm.² being passed over 8 minutes 20 seconds. Larger crystals are produced, as expected, at the lower current density. Additional to nucleation arguments, the vigorous rate of hydrogen evolution observed at 10 mA/cm.² is partially responsible for the decreased crystal size in this case.

Detailed Description Text (10):

The general applicability of electrolytically deposited phosphate coatings on metal substrates is further exemplified in FIG. 3, wherein scanning electron micrographs of the same coating (at two different magnifications) are as shown. The substrate in this case is titanium mesh on the stem of a commercially available Ti-6Al-4V hip

implant. This mesh provides an anchor for bony ingrowth into the implant. Strands of the mesh are the large cylindrical features in FIG. 3, which are 250 .mu.m in diameter. Crystallization was carried out at 1.0 mA/cm.sup.2 until 8 C/cm.sup.2 of charge had passed. The area used to determine the current density was based on the overall dimensions of the electrode, not the actual surface area of the mesh, which was not measured. All of the surfaces visible in FIG. 3 are coated and there are no discernable locations where preferential crystallization appears to have occurred. The morphology and physical integrity of calcium phosphate coatings produced by electrolytic deposition of the instant invention also may be varied using controlled potential electrolysis in order to produce the crystallization.

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Search Results - Record(s) 1 through 4 of 4 returned.

☐ 1. Document ID: US 6045683 A

L3: Entry 1 of 4

File: USPT

Apr 4, 2000

US-PAT-NO: 6045683

DOCUMENT-IDENTIFIER: US 6045683 A

TITLE: Modified brushite surface coating, process therefor, and low temperature conversion to hydroxyapatite

DATE-ISSUED: April 4, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Riley; Clyde	Huntsville	AL		
Kumar; Mukesh	Huntsville	AL		

US-CL-CURRENT: 205/318; 205/50

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWMC
Draw Desc	Image										

☐ 2. Document ID: US 5413693 A

L3: Entry 2 of 4

File: USPT

May 9, 1995

US-PAT-NO: 5413693

DOCUMENT-IDENTIFIER: US 5413693 A

TITLE: Electrocrystallization of strongly adherent brushite coatings on prosthetic alloys

DATE-ISSUED: May 9, 1995

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Redepenning; Jody G.	Lincoln	NE	68528	

US-CL-CURRENT: 205/318; 623/16.11, 623/901

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC
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☐ 3. Document ID: US 5330826 A

L3: Entry 3 of 4

File: USPT

Jul 19, 1994

US-PAT-NO: 5330826

DOCUMENT-IDENTIFIER: US 5330826 A

TITLE: Preparation of ceramic-metal coatings

DATE-ISSUED: July 19, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Taylor; Timothy E.	Huntsville	AL		
Riley; Clyde	Huntsville	AL		
Lacefield, Jr.; William R.	Birmingham	AL		
Coble; Harold D.	Huntsville	AL		
Maybee; George W.	Huntsville	AL		

US-CL-CURRENT: 428/216; 428/336, 428/469, 428/472, 428/621, 428/632, 428/655,
428/660, 428/704, 623/920

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMIC
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☐ 4. Document ID: US 5310464 A

L3: Entry 4 of 4

File: USPT

May 10, 1994

US-PAT-NO: 5310464

DOCUMENT-IDENTIFIER: US 5310464 A

TITLE: Electrocrystallization of strongly adherent brushite coatings on prosthetic alloys

DATE-ISSUED: May 10, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Redepenning; Jody G.	Lincoln	NE	68528	

US-CL-CURRENT: 205/318; 204/490, 623/901, 623/923

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KMIC
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L1 and (calcium adj1 \$phosphate)	4

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